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PROVISIONAL APPLICATION FOR PATENT COVER SHEET
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INVENTOR(S)			
Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)	
SUNGHO	JIN	SAN DIEGO, CALIFORNIA	
<input type="checkbox"/> Additional inventors are being named on the _____ separately numbered sheets attached hereto			
TITLE OF THE INVENTION (280 characters max) ARTICLES COMPRISING HIGH-ELECTRICAL-CONDUCTIVITY NANOCOMPOSITE MATERIAL AND METHOD FOR FABRICATING SAME			
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P19SMALL/REV05

Articles Comprising High-Electrical-Conductivity Nanocomposite Material and Method for Fabricating Same

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Field of Invention

The present invention relates to articles comprising composite materials. In particular, it provides articles, such as high power microwave devices, with strong, highly conductive nanocomposite materials for enhancing device performance.

Background of the Invention

High conductivity materials are important components in high power microwave systems (HPM systems) offering great potential for many civilian and military applications in the areas of communications and radar. For example, HPM systems can enable efficient and powerful microwave telecommunications or they can rapidly disrupt/damage enemy surveillance and communications hardware at significant standoff distances.

Practical HPM systems, however, are dependent on the realization of devices which are difficult to make. Two of the major technical barriers to realizing practical devices are the lack of high- current electron emitter cathodes and the RF breakdown of component materials. The intense high frequency RF electric and magnetic fields present in HPM devices are known to cause a mechanical and electrical breakdown on

surfaces and/or in volumes of the HPM device. In fact, such breakdown phenomena are believed to underlie the "pulse shortening" problem that has plagued HPM source studies for decades. See R. J. Barker and E. Schamiloglu, "High-Power Microwave Sources and Technologies", chapter 10 (IEEE Press, New York, 2001).

- 5 Surface fatigue from repeated pulsed heating on electron emission in the skin depth of HPM device wall component material, such as copper, can cause significant structural damage and can destroy the surfaces. The thermal shock caused by rapid temperature excursions between room temperature and the pulse heated temperature of even less than a few hundred degrees centigrade can induce defects and cracks in
- 10 the copper material with a resultant deterioration of performance. It is therefore desirable to increase the strength of the copper, or any equivalent conductor materials, used for the HPM wall component so that the material resists the thermal shock.

- Studies of strengthened copper materials for possible resistance to thermal fatigue and cracking problems in Cu in intense RF fields include investigation of Cu-
- 15 based composites containing Al_2O_3 dispersoid particles. See Paper # THD20, "The Use of Dispersion-Strengthened Copper in Accelerator Designs", by R. Valdiviez, et al. , International Linac Conference (LINAC 2000), Monterey, CA, 2000.)

- However, the use of insulating particles such as Al_2O_3 results in abrupt discontinuities in electrical conductivity that can produce local hot spots. Moreover, the
- 20 particles can reduce thermal conductivity. Additionally, insulating particles that reach the surface of the copper will provide localized sites of enhanced electric field in the vacuum.

Therefore, there is a need for a high-strength and fatigue-resistant material which is also highly electrically conductive, and preferably contains no electrically insulating particles.

Summary of the Invention

5 This invention discloses novel nanocomposite material structures which are strong, highly conductive, and fatigue-resistant. It also discloses novel fabrication techniques to obtain such structures. We describe three types of new nanocomposite materials comprising a high-conductivity base metal, such as copper, incorporating high-conductivity dispersoid particles so as to simultaneously minimize field
10 enhancements, maintain good thermal conductivity, and enhance mechanical strength. Use of metal nanoparticles with electrical conductivity comparable to that of the base automatically removes the regions of higher RF field and enhanced current density. Additionally, conductive nanoparticles will reduce the surface's sensitivity to arc or
15 sputtering damage, because if the surface becomes sputtered away to uncover the nanoparticles, their properties will not be dramatically different from the base surface. Most importantly, the secondary electron emission coefficients of all metals are small and close to unity, whereas insulating particles can give significant and undesirable electron multiplication.

Brief Description of The Drawings

20 The nature, advantages and various additional features of the invention will appear more fully upon consideration of the illustrative embodiments now to be described in detail with the accompanying drawings. In the drawings:

Fig. 1 describes an exemplary method of fabricating a nanocomposite containing highly electrically conductive particles via an electrolytic co-depositing process;

Figs. 2a, 2b and 2c are schematic illustrations of (a) base metal (prior art); (b) dispersion hardened high-conductivity nanocomposite material according to the invention; and (c) a functionally gradient surface structure, also according to the invention;

Fig. 3 illustrates an exemplary embodiment of electrical connectors incorporating high-conductivity, high-strength composite material according to the invention;

Figs. 4(a) and (b) represent embodiments of the invention with surface-concentrated nanocomposite structures that provide maximal fatigue resistance of the surface regions combined with good thermal dissipation of non-composite base material beneath surface;

Figs. 5(a) and (b) represent embodiments of the invention with (a) diamond film deposited over a base surface and a coated high work function layer, and (b) high thermal conductivity nanoscale diamond particles embedded in a base surface;

Fig. 6 is a block diagram describing an exemplary high power microwave system incorporating the high-conductivity, thermal-fatigue-resistant composite material according to the invention.

It is to be understood that the drawings are for purposes of illustrating the concepts of the invention and are not to scale.

Detailed Description of the Invention

In order to achieve a nearly ideal material for high power microwave devices, applicant has provided a high conductivity composite with mechanically-strengthening dispersoid particles that have electrical conductivity essentially matched with that of the base matrix metal. This invention can utilize any one of three types of dispersoids, the first being highly conductive metal nanoparticles artificially alloyed so that the matrix contains no or little dissolved element of the dispersoid metal, the second being dispersoid particles made of special, high-conductivity, intermetallic compound dispersoid nanoparticles, and the third being carbide, nitride or boride nanoparticles.

(a). Artificially Alloyed Nanocomposites

In traditional precipitation-hardened Cu alloys employing high temperature metallurgical reactions, the electrical conductivity of matrix metal [$\rho(\text{Cu}) \sim 1.67 \mu\Omega\text{-cm}$] is significantly reduced by the solute atoms that do not completely precipitate. Any alloying element remaining in solid solution in the Cu matrix deteriorates the electrical conductivity, often by an order of magnitude or more. In order to overcome such undesirable loss of conductivity in the Cu composites, applicant forms an "artificial alloy"

by incorporating high-conductivity metal nanoparticles of the second metal into the matrix metal using a process, such as electrodeposition, that incorporates the particles without dissolving them in the base. The use of electrodeposition to form composites comprising metal and dispersoid particles has been demonstrated previously, for example, using aluminum oxide dispersoid particles. See articles by J.C. Sadak and F.K. Sautter, "Ultrasonic Agitation Alters Microstructures and Properties of Electrodeposited Cobalt and Cobalt-Al₂O₃, Metals Engineering Quarterly, August 1974, page 44, and by J. L. Stojak and J. B. Talbot, J. Electrochem. Soc. Vol. 146, 4504 (1999). Such a use of insulator particles in copper is to be avoided for high power microwave devices as described earlier.

Fig. 1 schematically illustrates the electrodeposition of a composite 10 containing highly conductive metal nanoparticles 11 that are dispersed in the electroplating bath 12. The composite 10 is preferably copper based. The nanoparticles are incorporated in a controlled manner into the depositing Cu during the process. The most essential aspect of the invention is that the dispersoid metal is selected from very high conductivity metals such as Ag [$\rho \sim 1.59 \mu\Omega\text{-cm}$], Au [$\rho \sim 2.35 \mu\Omega\text{-cm}$], Al [$\rho \sim 2.65 \mu\Omega\text{-cm}$]. While these metal particles are not as strong as intermetallic compounds, the differences in lattice parameter, crystal structure, stacking fault energy, and dislocation movement behavior as compared to the host metal [such as Cu] impedes the motion of dislocations and mechanical slip or twinning deformation so that mechanical strengthening and fatigue-resistance are improved. Other metals with slightly lower conductivity such as Rh [$\rho \sim 4.51 \mu\Omega\text{-cm}$], Mo [$\rho \sim 5.20 \mu\Omega\text{-cm}$], W [$\rho \sim 5.65 \mu\Omega\text{-cm}$] can also be considered. While less preferred because of somewhat higher electrical

resistivity than Cu, these higher melting point metals still have reasonably high conductivity, and have other advantages in that they exhibit higher mechanical strength than non-refractory metals. Further, higher melting point metals have less tendency for undesirable dissolution into the Cu matrix during, for example, stress relief annealing or
5 baking to outgas any trapped gaseous elements incorporated during electrodeposition.

The desired elemental dispersoid particles 11 utilized in the inventive nanocomposites have electrical resistivity of less than $20\ \mu\Omega\text{-cm}$, preferably less than $6\ \mu\Omega\text{-cm}$, even more preferably less than $3\ \mu\Omega\text{-cm}$. The desired nanoparticle size is in the range of $\sim 5 - 500\ \text{nm}$ in average diameter, and preferably $10 - 100\ \text{nm}$ in average
10 diameter. In the inventive, high-conductivity nanocompositing process, the electroplating potential and deposition speed are carefully controlled toward a slower-rate process to minimize impurity or gas trapping. The nanoparticles 11 may optionally be pre-coated with a gold or other inert surface layer to minimize surface oxidation or contamination of the nanoparticles.

15 The nanocomposites so prepared may optionally be annealed, if desired, to relieve stress, to drive off trapped impurities, or to reduce any inadvertently formed oxide material. The desired annealing temperature is kept low enough to avoid significant diffusion of solute atoms into the matrix metal, which would cause deterioration of electrical conductivity of a Cu matrix. Hydrogen atmosphere annealing
20 at a relatively low temperature ($<\sim 400^\circ\text{C}$) is often sufficient to reduce copper oxide and to remove residual oxygen from Cu. Rapid thermal annealing at higher temperature is also an option. The desired volume fraction of the dispersoids in the inventive high-

conductivity nanocomposite is in the range of $\sim 0.2 - 20\%$, and preferably $0.5 - 10\%$, depending on the specific application.

Such high-conductivity nanocomposite materials are desirable for both high power microwave (HPM) devices operating in RF frequencies and for other applications.

- 5 For example, the materials can be advantageously used for electrical connectors and electrical contacts operating in DC or AC electrical operations. They provide both high mechanical strength to maintain spring force and good electrical contact.

Figs. 2(a), 2(b) and 2(c) schematically illustrate a conventional base material (Fig. 2(a)), and two forms of the "artificial alloy" structure (Figs. 2(b) and 2(c)). The base
10 material 20 is conventionally essentially free of the strengthening nanoparticles. The "artificial alloy" structure according to the invention can be deposited to form the basic device component structure having a substantially uniform distribution of the particles 21 throughout the volume as illustrated in Fig. 2(b), or with a gradient distribution of particles 21 as shown in Fig. 2(c).

- 15 Fig. 3 shows an exemplary electrical connector 30 where the connector pins 31 and mating spring connector slots 32 are made of the composite to exhibit high mechanical strength so as to maintain contact pressure for desired electrical conduction with minimal heating.

Alternatively, instead of the whole component being made of the dispersion-
20 hardened composite, only the surface of the component can be made to contain the dispersoid particles. Such a structure is schematically illustrated in Figs. 4(a) and 4(b). The co-electrodeposition of the base 20 and the dispersoid particles 21 is carried out to

form a relatively thin surface layer 40. For HPM components to be operated in a very high frequency RF environment, the thickness of the high electrical conductivity Cu nanocomposite layer can be slightly more than the RF penetration skin depth (e.g., ~ 1 μm thick coating would provide sufficient cushion for ~ 30 GHz operation for which the skin depth is ~ 0.4 μm .

For enhanced layer adhesion and improved resistance to thermal shock, an alternative embodiment of the invention calls for a functionally graduated microstructure in which the artificial alloy properties are gradually developed over a fraction of a micron, so as to avoid a sharp boundary, as illustrated in Fig. 4(b). The volume fraction of the dispersoid is altered as a function of electrodeposition time by a programmed nanocomposite electrodeposition process. The functionally gradient structure of Fig. 4(b) is particularly novel and practical in that the thermal and electrical properties are made to be in the optimal condition near the surface skin depth region of the composite layer while mechanical and structural continuity is maintained in relation to the base structure for enhanced reliability.

(b). **Nanocomposites with High-Conductivity Intermetallic Dispersoid**

Most intermetallic compounds exhibit high electrical resistivity in the range of ~ 10 – 100 $\mu\Omega\text{-cm}$. However, a few special intermetallics exhibit unusually low electrical resistivity, for example, $\text{Mn}_4\text{Al}_{11}$ [$\rho \sim 1.2$ $\mu\Omega\text{-cm}$], NiAl [$\rho \sim 1.0$ $\mu\Omega\text{-cm}$], and TiAl_3 [$\rho \sim 1.5$ $\mu\Omega\text{-cm}$]. See CRC Handbook of Electrical Resistivities of Binary Metallic Alloys, edited by K. Schroder, CRC Press, Boca Raton, FL, 1983, p. 90, 92, 97. According to the invention, these mechanically strong intermetallic particles are pre-made (e.g., by

atomizing, pulverizing or chemical precipitation) and incorporated into the base as by co-electrodeposition (Fig. 1) or by other processing. The resulting composite component exhibits a structure similar to those described in Figs. 2 and 4. In the case of high-conductivity intermetallic dispersoids or carbide/nitride dispersoids (discussed below), the stability of intermetallics is such that somewhat higher post-deposition annealing temperature, if needed, could be employed with a minimal solute dissolution into the matrix.

(c). **Nanocomposites Containing Conductive Carbides, Nitrides, or Borides**

Some carbides, nitrides, and borides exhibit high electrical conductivity and are insoluble in highly conductive base metals such as Cu. For example, cerium nitride [$\rho \sim 4.5 \mu\Omega\text{-cm}$] is insoluble in Cu. Other candidate carbide and nitride materials that can also be used as dispersoid nanoparticles in the inventive high electrical conductivity nanocomposites include TaC [$\rho \sim 40 \mu\Omega\text{-cm}$, thermal conductivity $K \sim 22 \text{ W/m.K}$], SiC [$\rho \sim 10^2 \Omega\text{-cm}$, $K \sim 250 \text{ W/m.K}$], ZrC, W_2C , TiC, TiN, and diamond nanoparticles including those doped to exhibit improved electrical conductivity.

Diamond exhibits the highest thermal conductivity of all known materials, about 5 times higher than Cu, so heat dissipation is enhanced resulting in the reduction of the temperature excursion that causes thermal shock during RF operation. Nanoscale carbon and graphite, such as carbon nanotubes, nanographite or nanocarbon particles have dimensions as small as a few nanometers, and hence can be efficient, conductive hardening dispersoids. These nanoparticles can be incorporated into nanocomposites

using the process shown in Fig. 1. The resulting composites can be made to exhibit the structures shown in Figs. 2 and 4.

In an alternative process for fabricating the inventive high-electrical-conductivity nanocomposites, the nanoscale particles (e.g. intermetallics, carbides, nitrides, borides, or diamond) are first coated with a relatively thick base metal (e.g., at least 0.1 μm , preferably at least 1 μm of Cu), and then the coated particles are pressed together and shaped into a desired component geometry. The shaped material is then sintered at a high temperature, e.g. 400 – 800°C preferably in an inert atmosphere or a reducing atmosphere such as hydrogen. The sintering temperature and time are advantageously controlled so that the diffusional mix of the elements in the particle and the base metal is minimized and thus the conductivity loss on alloying reaction is minimized. Yet another alternative processing is to mix the base metal powder with the nanoparticles (e.g. intermetallics, carbides, nitrides, borides, or diamond), consolidate and shape the mixture and sinter in a reducing atmosphere.

Figs. 5(a) and 5(b) illustrate embodiments of the invention using high thermal conductivity diamond. In Fig. 5(a), a diamond film 50 is deposited (as by CVD) over the base surface and coated with a layer of material 51 having a desired work function e.g. a high or low work function film. In Fig. 5(b), high thermal conductivity nanoscale diamond particles 52 are embedded in the base surface region, e.g., using the co-electrodeposition process of Fig. 1. The high thermal conductivity of diamond helps to laterally dissipate the heat generated by RF power present in HPM device operation, thus minimizing the temperature excursion range that the component experiences.

The nanoparticles to be electroplated into the base can be optionally pre-coated with a thin layer of base or other conductive metal to facilitate the co-electrodeposition process or adhesion with the electroplated matrix. For example, high conductivity intermetallic compound particles may be precoated with Cu, Ag, Au or Ni, using, for example, electroless plating prior to the co-electroplating process.

While the invention has been described herein primarily as a process for fabricating a strong, high conductivity copper-based material, the inventive process of co-electroplating or mixing to achieve very high conductivity composite can also be applied to many other base metals to obtain a high-strength, high-conductivity material. For example, the process can use base metals of Au, Ag, Ni, Co, Pd, Pt, Rh, Re, Cr, Zn, Au-Ag, or Cu-Ni.

Fig. 6 is an exemplary HPM system employing the novel materials described herein. First, a pulse power is supplied to the electron source which can be a hot cathode or a cold cathode. The electrons emitted from the cathode of the electron gun are then guided toward the RF converter where the RF signal is amplified with the electron beam. The RF signal or microwave signal then travels through the RF window and an antenna or an array of antennas transmit the microwave beam toward the target, for example, to disrupt the communication electronics and networks of enemy command center, or to send telecommunication signals toward the intended receiver. Advantageously, or more of the gun or converter comprise the strong, high conductivity nanocomposites described herein.

It can now be seen that one aspect of the invention is an article comprising a metal nanocomposite formed of a conductive base metal or alloy having high electrical conductivity and, dispersed within the base, nanoscale metal dispersoids of comparable high conductivity to strengthen the base without substantially reducing the conductivity of the composite as compared with the base. Typically the base material exhibits an electrical resistivity of less than 10 microhm-cm preferably less than 6 microhm-cm and more preferably less than 3 microhm-cm. The base is preferably copper but can advantageously be selected from Cu, Au, Ag, Ni, Co, Pd, Pt, Rh, Re, Cr, Zn, Au-Ag, and Cu-Ni.

The dispersoid particles can comprise conductive particles different from the base. The dispersoid particles may be elemental metal particles which are normally soluble in the base, but are incorporated in the base by an "artificial" structuring process (e.g. low temperature incorporation) such that the particles are not dissolved in the base.

Alternatively, the dispersoid particles can be non-elemental dispersoid particles which are insoluble in the base metal. They can be selected from high conductivity intermetallic compounds or from carbides, nitrides, borides, carbon, graphite or diamond.

The incorporation of the dispersoids can produce a composite material having mechanical strength enhanced by at least 30% over the base and high conductivity comparable to the base (less than 10 $\mu\Omega$ - cm, preferably less than 6 $\mu\Omega$ - cm and more preferably less than 3 $\mu\Omega$ - cm). The dispersoid can also improve the thermal fatigue

resistance by at least a 30% increase in the number of thermal cycles that can be endured.

The dispersoids can be incorporated throughout a body of the material or selectively near a surface. The concentration can be substantially uniform or on the
5 form of a gradient gradually decreasing in the direction from the surface to the interior so that mechanical adhesion, mechanical continuity and electrical continuity are not abruptly changed.

Another aspect of the invention is a method of fabricating a conductive nanocomposite material composed of a conductive matrix metal and nanoscale
10 conductive dispersoid particles. In one embodiment, material can be made by co-depositing the matrix metal and the dispersoid particles in an electrolyte solution. In another embodiment, the material can be made by coating high conductivity nanoscale particles with the matrix metal, pressing together and shaping the coated particles into a desired geometry, and sintering the shaped product in an inert or reducing atmosphere.

15 Alternatively, base metal particles and dispersoid particles can be mechanically mixed, the mixture can be pressed and shaped into desired form, and the shaped product sintered in an inert or reducing atmosphere.

Particularly useful articles employing the above-described nanocomposites include electrical connectors and high power microwave components.

20 It is understood that the above-described embodiments are illustrative of only a few of the many possible specific embodiments which can represent applications of the

invention. Numerous and varied other arrangements can be made by those skilled in the art without departing from the spirit and scope of the invention.

What is claimed is:

1. An article comprising an artificially alloyed metal nanocomposite such as based on Cu, Au, Ag, Ni, Co, Pd, Pt, Rh, Re, Cr, Zn, Au-Ag, and Cu-Ni, in which the base metal or alloy contains high electrical conductivity elemental metal particles which:

5

--- are soluble in the base matrix but are made insoluble by artificial structuring,

--- are selected from Ag, Au, Al, Pt, Pd, Mo, Co, Fe, Ni, B, Rh, W, Si, Zn,

--- exhibit an electrical resistivity of less than $10\ \mu\Omega\text{-cm}$, preferably less than $6\ \mu\Omega\text{-cm}$, even more preferentially less than $3\ \mu\Omega\text{-cm}$,

10

--- make the resultant artificial alloy composite material with an electrical resistivity of less than $10\ \mu\Omega\text{-cm}$, preferably less than $6\ \mu\Omega\text{-cm}$, even more preferentially less than $3\ \mu\Omega\text{-cm}$,

--- enhance the mechanical strength of the resultant artificial alloy composite material by at least 30 % over the base without the dispersoid particles.

15

--- improve the thermal fatigue resistance of the resultant artificial alloy composite material with at least 30 % increase in the number of thermal cycles that can be endured as compared to the base without the dispersoid particles.

2. The article of claim 1 where the high strength artificial alloy is in the form of surface layer on a matrix metal without dispersoid particles.

3. The article of claim 1 where the high strength artificial alloy is in the form of gradient structure with the dispersoid volume gradually decreasing from the surface to the interior so that mechanical adhesion, mechanical continuity and electrical continuity is not abrupt.

4. The structure of claim 1 where the high strength artificial alloy is made by co-electrodeposition of the matrix metal and the dispersoid particles in the electrolyte solution.

5. The article of claim 1 where the high strength artificial alloy is made by electroless or electroplating of the high conductivity particles with the matrix metal, pressing and shaping into desired component geometry, and then sintering in an inert or reducing atmosphere.

6. The article of claim 1 where the high strength artificial alloy is made by mixing the base metal particles with the high conductivity particles, pressing and shaping into desired component geometry, and then sintering in an inert or reducing atmosphere.

7. An article comprising a metal nanocomposite such as based on Cu, Au, Ag, Ni, Co, Pd, Pt, Rh, Re, Cr, Zn, Au-Ag, and Cu-Ni, in which the base metal or alloy contains high electrical thermal conductivity, non-elemental dispersoid particles which:

5 --- are insoluble in the base matrix metal,

--- are selected from a group of high electrical conductivity intermetallic compounds, such as Mn_4Al_{11} , NiAl, and $TiAl_3$, with electrical resistivity of less than $8\ \mu\Omega\text{-cm}$, preferably less than $3\ \mu\Omega\text{-cm}$, even more preferentially less than $1.5\ \mu\Omega\text{-cm}$, or from a group of carbide, nitride, boride, carbon, graphite, or diamond,

10 --- make the resultant composite material with an electrical resistivity of less than $10\ \mu\Omega\text{-cm}$, preferably less than $6\ \mu\Omega\text{-cm}$, even more preferentially less than $3\ \mu\Omega\text{-cm}$,

--- enhance the mechanical strength of the resultant composite material by at least 30 % over the metal without the dispersoid particles.

15 --- improve the thermal fatigue resistance of the resultant composite material with at least 30 % increase in the number of thermal cycles that can be endured as compared to the metal without the dispersoid particles.

8. The article of claim 7 where the high strength metal composite is in the form of surface layer on a matrix metal without dispersoid particles.

9. The article of claim 7 where the high strength metal composite is in the form of gradient structure with the dispersoid volume gradually decreasing from the surface to the interior so that mechanical adhesion, mechanical continuity and electrical continuity is not abrupt.

10. The article of claim 1 where the high strength metal composite is made by co-electrodeposition of the matrix metal and the dispersoid particles in the electrolyte solution.

11. The article of claim 1 where the high strength metal composite is made by electroless or electroplating of the high conductivity particles with the matrix metal, press and shape into desired component geometry, and then sinter at a high temperature in an inert or in a reducing atmosphere.

12. The article of claim 1 where the high strength metal composite is made by mixing the base metal particles with the high conductivity particles, press and shape into desired component geometry, and then sinter at a high temperature in an inert or in a reducing atmosphere.

13. An electrical connector device comprising the nanocomposite of any of claims 1-12.

14. A high power microwave device comprising the nanocomposite any of claims 1-12.

5 15. Method of fabricating the articles of any of claims 1-14 using co-electrodeposition, coated particle consolidation and sintering, or mixed particle consolidation and sintering.

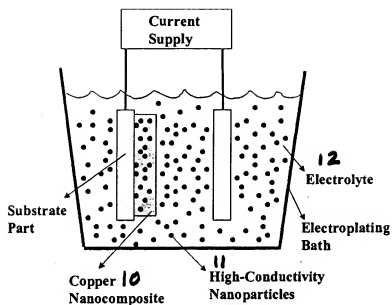


Fig. 1

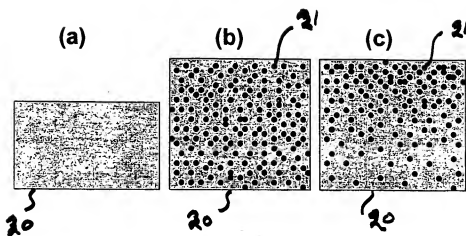


Fig. 2

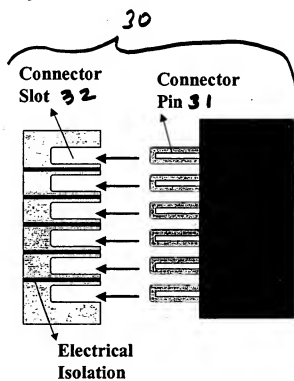


Fig. 3

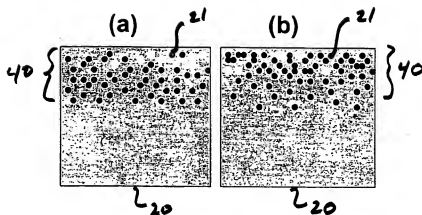


Fig. 4

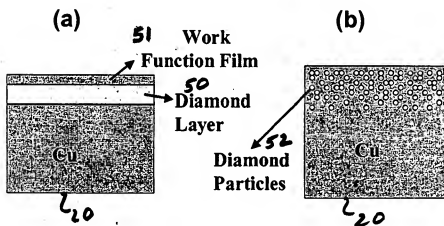


Fig. 5

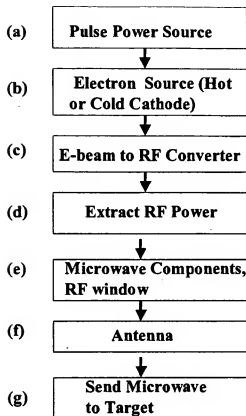


Fig. 6

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